

DECLARATION of HSUEH S. TUNG

I, Hsueh S. Tung, declare and state that:

1. I am a co-inventor of the subject matter described and claimed in US Patent Application No. 10/620,018 "Methods of Purifying Hydrogen Fluoride" (the '018 Application").
2. I hold an undergraduate degree in Chemical Engineering from Taipei Institute of Technology (1970) and a PhD in Chemistry from Michigan State University (1981). I have been employed by Honeywell International, Inc., and its predecessor(s) in interest, the assignee(s) of the application, since 1981, holding various positions including Chemist and Senior Project Leader. I am currently a Technical Manager for the Fluorocarbon Process Technology Group. I have extensive experience and familiarity with the development of methods for preparing and purifying anhydrous hydrogen fluoride (HF).
3. In furtherance of the prosecution of the '018 Application, I have supervised the experiments described below which illustrates the recovery of anhydrous HF from a mixture of HF and HFC-245fa using 80 wt. % sulfuric acid *without* using flash distillation.
4. To conduct this experiment, the procedure described in Example 1 of the '018 Application was repeated, except that no flash distillation was performed before fractionation of the HF from the HF/sulfuric acid mixture. More specifically, a mixture consisting of about 75 wt. % HFC-245fa and about 25 wt. % HF was vaporized and fed to the bottom of a packed column at a feed rate of about 2.9 lbs per hour for about 4 hours. A stream of about 80 wt. % sulfuric acid (i.e., 80/20 H₂SO₄/H₂O) with about 4% HF dissolved therein was fed continuously to the top of the same packed column at a feed rate of about 5.6 lbs per hour during the same time frame. A gaseous stream exited the top of the column that was determined to comprise HFC-245fa with less than 1.0 wt. % HF therein. The concentration of HF in the sulfuric acid in the column bottoms was determined to be about 14.5 wt. %. Instead of flashing, subsequent desorption of HF was conducted in a fractionation (i.e., distillation) column with a reboiler temperature of about 110 – 140 °C. About 304 grams of anhydrous HF was recovered as a distillate. This distillate was determined to have a sulfur content of 313 ppm.
5. Example 1 of the '018 Application shows that under similar test conditions, a combination of flashing followed by column distillation results in a distillate having a sulfur content of about 43 ppm.

6. The data of the experiment demonstrates that, using a low concentration sulfuric acid technique, column distillation alone (i.e., without a flashing step) results in higher levels of sulfur impurities compared to a combination of flashing followed by column distillation. One skilled in the art would *not* have known or expected that subjecting a mixture of hydrogen fluoride and dilute sulfuric acid to the *combination* of a flash distillation process followed by a fractionation process would dramatically reduce the amount of sulfur impurities in the process stream. To the contrary, one skilled in the art would have expected that a simple column fractionation alone could be used to recover substantially pure anhydrous hydrogen fluoride from sulfuric acid (i.e., anhydrous hydrogen fluoride having very low sulfur content), just as we expected before our discovery. More particularly, one would expect to see substantially lower sulfur impurities in the distillate of anhydrous HF after a simple fractionation due to the large difference between the normal boiling point of HF (20 °C) and that of sulfuric acid (338 °C).
7. However, as the experiment above shows, we found this not to be the case. That is, instead of low sulfur levels, we found that a relatively high level of sulfur impurities remained in the anhydrous HF distillate after column fractionation. After substantial experimentation with different distillation techniques, we carried out a process which demonstrated that flashing the HF and sulfuric acid mixed stream *followed by* fractionation is capable of dramatically decreasing the level of sulfur impurities compared to fractionation alone. This result was unexpected. It is our theory that sulfuric acid decomposes to some unknown sulfur compound having a boiling point less than that of HF, and as a result, this sulfur compound was found in the distillate of anhydrous HF that was processed by column fractionation alone. We believe that flash distillation followed by fractionation minimizes this decomposition.
8. I hereby declare that all statements made herein of my knowledge are true and that all statements made on information and belief are believed to be true and that any willful false statements may jeopardize the validity of the application or any patent issued thereon.

Hsueh Sung Tung
Hsueh Tung

11/20/07
Date